ADMINISTRATIVE INFORMATION

1. **Project Name:** Development of Ultrananocrystalline Diamond Coatings

2. **Lead Organization:** Argonne National Laboratory
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Argonne, Illinois, 60439

3. **Principal Investigator:** John N. Hryn, Ph.D.
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4. **Project Partners:** ADT – Advanced Diamond Technologies, Inc.
POC: Mr. Neil Kane (312) 404-3507
John Crane, Inc.
IPLAS Innovative Plasma Systems
Morgan Advanced Ceramics
Northwestern University
University of Illinois at Chicago

5. **Date Project Initiated:** October 1, 2001

6. **Expected Completion Date:** September 30, 2007

PROJECT RATIONALE AND STRATEGY

7. **Project Objective:** (Please provide 1-2 sentences describing the objective of this project.)

The objectives of this six-year R&D project, which falls under the "knowledge base or core activities R&D" in the *Industrial Materials for the Future* (IMF) program plan, are to (a) understand the fundamental processes involved in the growth of ultrananocrystalline diamond (UNCD) coatings, (b) develop a technological base for UNCD applications, and (c) demonstrate the applicability of UNCD coatings in industrial applications, such as in multipurpose mechanical pump seals.

8. **Technical Barriers Being Addressed:** (Please provide 1-3 sentences describing the problems and/or barriers limiting industrial energy efficiency which this project is addressing.)

Increased corrosion resistance and improved wear resistance of industrial materials could substantially improve industrial energy efficiency. Ultrananocrystalline diamond (UNCD) is diamond: the hardest, most-wear-resistant, and most corrosion-resistant material. This project will strengthen the fundamental understanding and processing methods of UNCD coatings that, when applied to industrial materials, will increase their corrosion resistance and improve their wear resistance. Sliding applications of UNCD-coated materials, such as in multipurpose mechanical pump seals, offer the greatest potential for improved energy efficiency in industry. Four technical barriers being addressed to allow wide-spread use of UNCD coatings in industrial applications are:

- Nucleation of UNCD during the coating process
- Effect of initial surface roughness of the substrate on the friction coefficient of UNCD coatings
- Adhesion of the UNCD coating to the substrate in service
- Scale-up of UNCD deposition process to allow cost-effective coating of materials

9. **Project Pathway:** (Please provide a one-paragraph summary of the approach, or pathway, being used to address the barriers. Emphasize the overall strategic approach for the project, not individual R&D tasks.)

The approach taken in this project is to first understand the UNCD plasma deposition process, including the seeding requirements to promote the initial nucleation needed to grow uniform UNCD coatings. This is followed by understanding and developing UNCD processing methods to insure the effects of surface roughness and adhesion are well understood. Finally, feasibility of scaling up the technology needs to be demonstrated by coating sufficient UNCD-coated seals to allow for field testing. As fundamental understanding and processing methods of UNCD coatings mature to the point at which an industry-specific or cross-cutting technology application appears viable (such as application in rotating pump seals), further development of that application will be more appropriate in the industry-specific or crosscutting technology portions of the IMF subprogram. A company, Advanced Diamond Technologies, Inc., has been formed to further develop, commercialize, and market the technology.

10. **Critical Technical Metrics:** (Please indicate how success or failure will be measured for this project by stating the baseline technical metric(s) and the metric(s) needed for realization of the project objectives.)

Success or failure of this project will be measured by our ability to meet our 3 project objectives. Specifically, we have made substantial progress in understanding how UNCD coatings nucleate and grow, and have completed the work on plasma R&D on the 6-inch IPLAS reactor, and on UNCD seeding and nucleation. We have developed a solid technological base for UNCD applications that we are now extending to the 11-inch IPLAS reactor. Scaling up the UNCD coating technology is critical to our ability to coat materials for industrial applications in a cost-effective manner. Finally, and most importantly, we need to demonstrate the energy and value-added benefits of UNCD coating in industrial applications. To this end, we need to conclude this project with a successful field test of UNCD-coated seals in multipurpose mechanical pumps. The critical metric for industry will be the ability of UNCD-coated seals to perform at an overall lower cost compared to current practice.

PROJECT PLANS AND PROGRESS

11. **Past Accomplishments:** (Please summarize the major accomplishments and **key** milestones achieved to date. Note: May not be applicable for projects initiated in FY04.)

Plasma R&D

- New 6-inch IPLAS microwave plasma system installed and commissioned
- Completed R&D on 6-inch unit, optimized process, demonstrated production proof-of-concept
- New 11-inch IPLAS unit (50% co-funded by DOE-OBER) was installed

Surface Seeding Study

- Completed surface seeding study, investigated CDC – found to be not beneficial when used in conjunction with UNCD
- Identified effect of Fe contaminants in SiC to UNCD growth
- Developed new seeding methodology

UNCD Materials and Processing Development

- Demonstrated growth of UNCD on a variety of seal substrate materials (alpha SiC, reaction-bonded SiC, WC, CDC – includes design and commissioning of CDC facility)
- Demonstrated UNCD growth on multiple 1-inch seals simultaneously, with required flatness and roughness, uniform thickness, and minimal waviness

Pump Seal Application

- Solved delamination issue, adhesion of UNCD-to substrate determined
- Identified testing protocol
- Broader industrial partner base established
- Launched ADT to be commercialization vehicle for UNCD

12. **Future Plans:** (Please summarize the **key** milestones and deliverables with dates for the life of this project. A comprehensive activities schedule is not required.)

Plasma R&D	
Commission and optimize 11-inch IPLAS system	9/05
Design and construct automated system	6/07
UNCD Materials and Processing Development	
Demonstrate UNCD coating of multiple seals using 11 inch IPLAS	3/05
Pump Seal Application	
Verify tribological benefit of UNCD coated seals	9/04
Long-term pump tests	9/05
Complete field tests and analyses	6/07
Other applications	
Demonstrate UNCD coatings on gas seals	9/05
Evaluate UNCD coated gas seals (field tests)	6/07
Final Report	9/07

13. **Project Changes:** (Please describe changes in scope, approach or schedule during the past year in response to any unforeseen problems/issues or successes.)

Two changes to the project occurred over the past year. First, we have uncovered an effect of initial surface roughness of the substrate on the friction coefficient of UNCD seals. This effect needs to be thoroughly understood, since a significant portion of the benefits from using UNCD arise from energy savings due to reduced friction losses. A subtask to verify the tribological benefit of UNCD was added to the milestone plan. Secondly, the project scope was extended to include gas seals -- seals designed to draw air into the gap between mating surfaces in rotating seals, thus minimizing friction losses.

14. **Commercialization Potential, Plans, and Activities:** Describe the end-use application and market potential for the project, and the plans, progress, and partners for commercial application/adoption, where appropriate; identify what the product of the project will be and how this product will be introduced/disseminated to industry.)

A company, Advanced Diamond Technologies, was formed by Argonne National Laboratory to be the commercialization vehicle for UNCD. ADT has, over the past year, developed partnerships and entered into collaborations with leading pump seal companies who, like our project partner John Crane, Inc., will be marketing partners of ADT. Through ADT, a mechanism exists for rapid, and broad, deployment of UNCD technology to a variety of IOF (and non-IOF) industries.

In the case of pump seals, ADT will be a toll-coating provider to John Crane, Inc. It is envisioned that Crane will provide engineered seals to ADT and ADT will add the UNCD coating. With this business arrangement, ADT is free to focus on its competency which is the production of large area UNCD coatings, while leveraging the unmatched marketing and distribution capabilities of Crane worldwide. This arrangement was optimized around speed to market.

ADT has already done the financial modeling to provide justification to the assertion that the UNCD platform can be used to provide cost-effective and market-ready coated seals. Advanced Diamond Technologies was judged "Most Promising Company" at the Nanotechnology Venture Fair in

La Jolla, California in September 2002. An R&D 100 award, entitled "Large-Area Deposition of Ultrananocrystalline Diamond Films" was won in 2003.

15. **Patents, Publications, Presentations:** (Please list number and reference, if applicable. If more than 10, please list only 10 most recent.)

    <u>6 Presentations and 1 Publication: June 2003-May 2004</u>

    Kovalchenko, A.; Elam, J.; Auciello, O.; Hryn, J.; Gruen, D.; Erdemir, A.; Carlisle, J., "Improved adhesion of ultrananocrystalline diamond coatings to SiC mechanical seal surfaces," The Society of Tribologists and Lubrication Engineers 2004 Annual Meeting & Exhibition, Toronto, Canada; May 17-20, 2004 (in preparation).

    Zinovev, A. V.; Elam, J. W.; Moore, J. F.; Hryn, J.; Auciello, O.; Carlisle, J.; Pellin, M. J., "Coating of SiC surface by thin carbon films with using the carbide-derived carbon (CDC) process," International Conference on Metallurgical Coatings and Thin Films; San Diego, CA; Apr 19-23, 2004.

    Kovalchenko, A.; Elam, J.; Auciello, O.; Hryn, J.; Gruen, D.; Erdemir, A.; Carlisle, J., "Improved adhesion of ultrananocrystalline diamond coatings to SiC mechanical seal surfaces," USDOE International Conference on Metallurgical Coatings and Thin Films (ICMCTF 2004) ; San Diego, CA; Apr 19-23, 2004.

    Zinovev, A. V.; Moore, J. F.; Hryn, J. N.; Auciello, O.; Carlisle, J. A.; Pellin, M. J., "Chemical bonding and morphology of thin carbon films grown on SiC surface via the carbide-derived carbon (CDC) process," The Metallurgy Society 2004 TMS Annual Meeting and Exhibition ; Charlotte, NC; Mar 14-18, 2004.

    Zinovev, A. V.; Moore, J. F.; Hryn, J. N.; Auciello, O.; Carlisle, J. A.; Pellin, M. J., "Chemical etching of silicon carbide ceramic surfaces in chlorine-containing gas mixtures," American Ceramic Society 28th International Cocoa Beach Conference and Exposition on Advanced Ceramics and Composites; Cocoa Beach, FL; Jan 25-30, 2004.

    Zinovev, A. V.; Moore, J. F.; Pellin, M. J.; Carlisle, J. A.; Auciello, O. H.; Hryn, J. N., "XPS study of chemical conversions on silicon carbide surface treated in chlorine-containing gas mixtures," 10th International Conference on Silicon Carbide and Related Materials; Lion, France; Oct 5-10, 2003.

    Zinovev, A. V.; Elam, J. W.; Moore, J. F.; Hryn, J. N.; Auciello, O. H.; Carlisle, J. A.; Pellin, M. J., "Analysis of Thin Carbon Films Prepared on SiC Surfaces Using the Carbide-Derived Carbon Process", <u>Thin Solid Films</u>, (submitted), Feb. 2004.

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